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systems with different stabilizers may be used. The degree of reactions during the sintering significantly vary depending on the particle size, particle size distribution, and densification temperatures. X-ray diffraction was used to determine any detectable chemical reactions during the sintering.

HIPing of zirconia is a well established method for eliminating strength limiting pores or cavities. The flexural strength of sintered ceramics are often improved up to 50% compared to that of pressureless sintered ceramics resulting in a major gain in their structural reliability and machinability. Other resistivity modifiers may have a limited reaction with Y-TZP. However, the modifiers may be thermochemically reduced during the typical HIPing in inert gas, e.g., argon or the like, using graphite heating elements. It is well known that such HIPing may thermochemically reduce such compounds releasing gaseous products. Such gas forming reactions limit the densification caused by bloating, swelling, and/or cracking. For instance, most transition oxides, such as Fe_2O_3 , Co_2O_3 , NiO , and TiO_2 , suffer from such reactions. Accordingly, it is very desirable to find a thermochemically stable, conductivity modifier to avoid such problems. Alternatively, an oxygen HIPing may be used to avoid such negative reactions. However, the high cost of oxygen HIPing is often undesirable for the manufacture of cost effective products.

A number of different ESD dissipation applications require various resistivities ranging from 10^3 to 10^{11} Ohm-cm. The conductivity of ceramics can be controlled by adjusting the amount of conductivity modifiers and/or using different ratios of particle sizes of conductivity modifier over base material. Accordingly, various ceramic compositions may be prepared to cover a variable range of conductivities. Therefore, it is very desirable to establish a method to tune (or tailor) the resistivity of a composition by a rather simple heat treatment (or annealing) of ceramics in air or gas atmosphere. In most ceramic systems the resistivity can change depending on the density of charge carriers such as interstitial cations or vacancies.

It is known that a different heat treatment of ceramics at high temperature in a known atmosphere from their thermal history (densification temperature and atmosphere) can change the equilibrium density of charge carriers in ceramic bodies resulting in a different resistivity. The degree of change in a given system has not been studied for most ceramic systems. In addition, various minor impurities in the system may significantly influence such changes. Further, there may be a few different charge carriers active in some ceramic systems. Accordingly, it is very important to establish the control of resistivities by experiments.

Most ESD dissipative ceramics tools require machining into precision shapes and dimensions. Therefore, if there is a compositional gradient on the surface of ceramic body the ESD dissipating characteristics may vary.

It was found that the volume and surface resistivity fall within an order of magnitude from a sample. Further, the voltage decay time correlates rather well with the surface resistivity in homogeneous material. For instance, a sample with a low surface resistivity (e.g., $R_s=10^6$ Ohm/sq.) exhibits a short voltage decay time, i.e., ~50 ms. If there is a macroscopic compositional segregation in the composite, the dissipation behaviors of material may vary depending on the compositional fluctuation.

Characterization Methods:

Densities of ceramics were measured by the water displacement method with temperature and atmospheric pressure corrections.¹²

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Microstructure of ceramics were observed by optical and scanning electron microscopes on polished cross-section of samples prepared by a standard polishing procedure.

Color of ceramic pieces were measured by Miniscan XE⁵ Plus available from Hunter Associates Laboratory, Inc., Reston, Va., using 45°/0° optical geometry and 7.6 mm aperture.

X-ray diffraction (XRD) method was used to determine crystalline phases of powders and densified ceramics.

Young's modulus, shear modulus and Poisson's ratio of ceramics were measured by a pulse-echo method using ultrasonic transducers.

Hardness of ceramics were determined by Vickers hardness method at loadings of 5 to 20 Kgf.

The 4-point flexure strength of ceramics were determined by a method specified in ASTM C1160-90 using 3x4x50 mm type-B specimen. Assuming a Weibull modulus of about 10, it is estimated that this method reports a flexural strength²⁰ that is about 20% lower than that found using JIS R 1601-81 (3-point flexure test using 30 mm span). Average strength of 5 to 15 bars were reported.

Fracture toughness of ceramics were determined by an indentation crack length method using Vickers indenter at loadings of 10 to 40 Kgf. An indentation crack strength method was also used in some occasions.

Volume and surface resistivities (DC) were determined according to methods specified by the ASTM¹³, and others at 10 or 100 V. The surface of ceramics was ground by a diamond wheel and cleaned in a ultrasonic bath prior to the resistivity measurement. As an electrode, type 1215 silver-loaded conductive rubber sheet about 0.55 mm thick made by Chomerics Inc. of Woburn, Mass., was used.

Voltage decay time is a measure of charge dissipation and is determined by methods described in literature.^{14,15,16,17}
Uses of Semi-Insulating, ESD Dissipative Materials:

The following is a partial list of the many possible uses of the ESD dissipative materials of the present invention. These suggested uses include the following:

- MR (magnetoresistive) head substrates,
- Transfer tools for magnetic head manufacturing (lapping, polishing, cleaning),
- Tools, fixtures, and containers for HGA (head gimbal assembly), HSA (head stack assembly), HDD (hard disk drive) assembly,
- Tools, fixtures, and containers for wafer handling, processing, cleaning in water, solvents, and with carbon dioxide,
- Tools, fixtures, and containers for wire bonding, trimming, cutting,
- Pick-and-place nozzles for IC chip handling,
- Nozzles for adhesive and solder dispensing,
- Wafer handling fixtures, end effectors, vacuum chucks,
- IC handling/test fixtures,
- Tools, fixtures, and containers for ESD sensitive devices, e.g., CMOS, GaAs, SiGe, InP, and MRAM, MEMS
- Assembly fixtures for IC's and magnetic heads,
- Personal tools: tweezers, screw drivers, scissors, blades,
- Parts for robotic fingers,
- Tools, fixtures, and containers for electro-optic coating process,
- Tools, fixtures, and containers for reticles (photo mask), and the like.

In fabricating the component parts described above (which is a non-exhaustive list) the ESD dissipative materials of the present invention may be employed as the entire

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component part, or as a portion thereof. For example, ESD dissipative materials may be shaped into the various components by grinding, machining, or the like—in order to have a component part this is entirely formed from an ESD dissipative material. On the other hand, small portions of the component parts may be all that require an ESD dissipative material to be added thereto. In that case, one or more small machined or ground (or otherwise formed) segments of the ESD dissipative materials of the present invention (note—two or more materials may be combined if desired) would be added to the component part (either permanently or in a replaceable manner) in the critical area requiring ESD protection. Persons having ordinary skill in each respective component art can readily adapt the ESD dissipative materials of the present invention to the specific requirements of each component listed herein, as well as others not recited above.

The present invention will be further illustrated with reference to the following examples which aid in the understanding of the present invention, but which are not to be construed as limitations thereof.

EXAMPLE 1

ZnO

A Y-TZP powder containing about 2.8 mol % yttria (YZ110) available from Saint-Gobain Ceramics & Plastics (SGC&P) was mixed in water with varying amounts of zinc oxide available from Zinc Corporation of America in a jar mill with Y-TZP milling media. The surface areas of YZ110 and ZnO powders were 7 and 20 m²/g, respectively, prior to milling. The milled powder was granulated by a laboratory method to pressable powder. The powder was uniaxially pressed into steel molds at 40 MPa, and then cold isostatically pressed (or "CLPed") at 207 MPa to form green bodies.

The green bodies were sintered at 2° C./min to a desired sintering temperature (1350°–1500° C.) for 1 hr to achieve a sintered density of greater than 97% TD (theoretical density). The theoretical density was calculated by a mixing rule based on volume % of ingredients assuming no reaction between two phases during the sintering. Some sintered bodies were HIPed in Ar at 1300° C. for 45 min to achieve the full density. All samples were HIPed to greater than 99.5% TD., mostly greater than 99.8% TD.

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The resulting densified bodies by sintering and sinter-HIPing were measured for density, Young's modulus, hardness, strength, fracture toughness, phase microstructure, volume and surface resistivities, and ESD dissipation (voltage decay) as summarized in Tables 5 and 6.

Effect of ZnO Content and Grain Size:

Table 5 summarizes volume resistivities as a function of vol. % ZnO in Y-TZP of pressureless sintered samples. It is noted that the resistivities for the ESD dissipation ($10^3 < R_v < 10^{12}$ Ohm-cm) can be achieved by composite materials with 15–35 vol. % ZnO and 85–65 vol. % Y-TZP. These sintered materials can be prepared to greater than 99% T.D. at 1400°–1450° C. for 1–2 Hr. in air.

Sample No. 16 was prepared with a Y-TZP powder containing 3 mol % yttria (HSY3.0) and sintered at 1250° C., which is 150° C. lower temperature than others, to minimize the grain growth of ZnO. It is noted that the volume resistivity of this sample is 5 orders of magnitude lower than Sample No. 2. The present result indicates the fine grain size of conductive phase (ZnO) by low temperature sintering is favorable to achieve a lower resistivity. Further, it is believed that the amount of modifier could be lower by low temperature sintering and reduced grain growth to achieve the same ESD dissipative resistivity.

25 Effect of Annealing Temperature and Atmosphere:

Surprisingly, the resistivity of Sample No. 8 after HIPing at 1350° C./207 MPa for 45 min showed a significant reduction from 7×10^{11} to 5×10^3 Ohm-cm. Samples No. 9–14 show the effect of heat treatment (annealing) in air on the resistivity of HIPed 20 vol. % ZnO/YZ110 composites showing the tunability of resistivity by a simple heat treatment. These results further indicate that it is not necessary to prepare various compositions to cover a broad range of resistivities for various ESD dissipative applications. 35 Annealing at different atmospheres (Sample No. 15) also provide additional means to achieve the tunable resistivity with this composition.

An over-annealed sample with R_v of 5×10^{11} Ohm-cm was annealed at 890° C. for 5 Hr. in 2% H₂/N₂ atmosphere, 40 resulting in a resistivity of 5×10^6 Ohm-cm. This result indicates that the resistivity control of this material is reversible. Accordingly, it is believed that the desired resistivity can be achieved by composition, annealing in air, and/or annealing in a controlled atmosphere.

TABLE 5

ID #	Zirconia type	ZnO (Vol %)	Densification (Method/Temp.)	Heat treatment		Rv (Ohm-cm)
				(Temp/time)	(Atm.)	
1	YZ110	15	S/1400° C.*	—	—	2.0E12
2	YZ110	20	S/1400° C.	—	—	7.4E11
3	YZ110	25	S/1400° C.	—	—	5.6E10
4	2.6YTZP	25	S/1400° C.	—	—	7.6E09
5	2.6YTZP	30	S/1400° C.	—	—	9.1E07
6	YZ110	30	S/1400° C.	—	—	7.9E06
7	2.6Y-TZP	35	S/1400° C.	—	—	6.9E04
8	YZ110	20	H/1350° C.**	—	—	5.0E03
9	YZ110	20	H/1350° C.	560° C./5 Hr	Air	4.0E04
10	YZ110	20	H/1350° C.	585° C./5 Hr	Air	9.0E04
11	YZ110	20	H/1350° C.	600° C./5 Hr	Air	1.7E06
12	YZ110	20	H/1350° C.	630° C./5 Hr	Air	5.0E06
13	YZ110	20	H/1350° C.	680° C./5 Hr	Air	1.3E11
14	YZ110	20	H/1350° C.	630° C./24 Hr	Air	2.5E11
15	YZ110	20	H/1350° C.	810° C./2 Hr	2% H ₂ /N ₂	6.0E06
16	HSY3.0	20	S/1250° C.			2.9E06

S*: pressureless sintering in air, H**: HIPing in argon

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Transient Current and Voltage Decay Time:

Samples of composition 20% and 25% ZnO/YZ110 were prepared and were heat treated in air at temperatures in the range 600°–650° C. to give the resistivity values shown below. Measurements of transient current were made with the apparatus shown in FIG. 1.

To make a measurement, the plate (which is part of the charged plate monitor) is charged to a fixed voltage. This loads the plate with about 7 nC of charge at 350 V. This charge is then dissipated to ground by touching the sample with the movable contact. The peak transient current is determined from the peak voltage seen from the current probe by the oscilloscope, which records voltage every nanosecond.

Decay time is measured by reading the voltage output of the charged plate monitor on a digital oscilloscope as a function of time. The time for the voltage to fall from 900 V to 100 V is measured from the oscilloscope after initial charging to 1,000 V. Instrument electronics set a limit of 25 ms as the shortest measurable decay time. The following results were obtained:

TABLE 6

Sample	Volume resistivity (Ohm-cm)	Peak current at 400 V (mA)	Peak current at 500 V (mA)	Decay time (ms)
#18 (20% ZnO)	7.7 × 10 ⁵	1.6	16	less than 25
#10 (20% ZnO)	2.2 × 10 ⁶	less than 1	14.7	less than 25
#11 (20% ZnO)	1.6 × 10 ⁸	Not measured	1	43
#34 (25% ZnO)	3.1 × 10 ⁹	less than 1	2.0	Not measured

Table 7 summarizes properties of 20 vol. % ZnO/YZ110 composition after HIPing. The material is highly dense with a relative density of greater than 99.5% T.D. with a light yellow color (lighter than 50% of 16 gray scale), mechanically strong with a flexural strength of the material of 1 GPa, substantially safe with low transient current, and highly dissipative with a decay time from 1,000V to 100V is typically less than 50 ms. Accordingly, the material should be good for the ESD dissipative ceramics. Further, the residual magnetic flux density of this material is substantially negligible due to the absence of Fe, Co, Ni, and Cr-oxides in the composition.

TABLE 7

Property Name	SI Units	(Mean values)	Test method
Color		Light yellow-	Visual/color meter
Density	g/cm ³	5.99	Water displacement
Porosity	%	less than 0.5%	Density and polished surface
Grain size	µm	1	Linear intercept
Young's Modulus, 20° C.	GPa	195	Ultrasonic wave velocity
Hardness - Vickers, 20° C.	GPa	9.2	Vickers hardness at P = 20 Kgf
Flexural Strength, 20° C.	MPa	1000	4-point bending, ASTM-C1161-90
Fracture Toughness, 20° C.	MPa	5.9	Indentation crack length
Fracture Toughness, 20° C.	MPa	4.2	Indentation strength at P = 40 Kgf

EXAMPLE 2

SnO₂

A Y-TZP powder (HSY3.0) containing about 3 mol % yttria available from Zirconia Sales of America was mixed with varying amount of tin oxide (SnO₂, T1186)¹⁸ available

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from SGC&P in a jar mill with Y-TZP milling media. The powder has nominal dopants of 1% Sb₂O₃ and 0.5% CuO for improved conductivity. Prior to the mixing the doped tin oxide was milled to surface area of ~15 m²/g. Surface area of Y-TZP was 7 m²/g prior to milling. Another commercial tin oxide powder (available from Alfa Aesar, Ward Hill, Mass.) milled to a surface area of 12 m²/g was also used. The milled powder was granulated by a laboratory method to pressable powder. The powder was uniaxially pressed into steel molds at 40 MPa, and then CIPed at 207 MPa to form green bodies. The green bodies were sintered at 2° C./min to a desired sintering temperature (1400–1500° C.) for 1 hr to achieve a sintered density of greater than ~95% T.D. The theoretical density was calculated by a mixing rule based on the composition assuming no reaction between two phased during the sintering. Some sintered bodies were HIPed in Ar at 1400° C. for 45 min to achieve the full density.

The resulting densified bodies by sintering and sinter-HIPing were measured for density, Young's modulus, hardness, strength, fracture toughness, phase microstructure, volume and surface resistivities, and ESD dissipation (voltage decay).

The pure SnO₂ is an insulative material. It was confirmed that compositions of 10–50% SnO₂/Y-TZP are insulative, Rv=10¹³ Ohm-cm, as shown in Table 9. A limited heat treatment in Ar and N₂ in a tube furnace did not show a significant decrease in the resistivity. The color was remained as light grayish, lighter than 50% of 16 gray scale. A HIPed sample at 1400° C./207 MPa/60 min in Ar turned into a medium gray and showed resistivities of 10⁷ Ohm-cm.

TABLE 9

	SnO ₂ (Vol %)	Densification (Method/Temp.)	Rv (Ohm-cm)
Undoped	10	S/1450° C.*	1.2E13
Undoped	20	S/1450° C.	1.5E13
Undoped	30	S/1450° C.	3.7E13
Undoped	35	S/1450° C.	3.4E13
Doped	50	S/1450° C.	6.5E12
Doped	50	H/1350° C.**	6.1E07
Doped	50	H/1300° C.**	2.6E07

S*: pressureless sintering in air, H**: HIPing in argon

Properties of HIPed 50% doped-SnO₂/HSY3.0 were measured as summarized below. The material shows light to medium grey. The material is stiffer and harder than those of 20 vol. % ZnO/YZ110 which are similar to those of typical Y-TZP's. In addition, the material is also substantially non-magnetic.

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TABLE 10

Property Name	SI Units	(Mean values)	Test method
Color		Light gray	Visual/color meter
Density	g/cm ³	6.43	Water displacement
Porosity	%	less than 1%	Density and polished surface
Grain size	µm	1	Linear intercept
Young's Modulus, 20° C.	GPa	224	Ultrasonic wave velocity
Hardness - Vickers, 20° C.	GPa	11.4	Vickers hardness at P = 20 Kgf
Flexural Strength, 20° C.	MPa	650	4-point bending, ASTM-C1161-90
Fracture Toughness, 20° C.	MPa m ^{1/2}	3.9	Indentation crack length
Fracture Toughness, 20° C.	MPa m ^{1/2}	3.9	Indentation strength at P = 40 Kgf

EXAMPLE 3



A batch of LaMnO_3 was prepared by the solid-state reaction of an equimolar powder mixture of La_2O_3 and Mn_2O_3 at 1400° C. for 2 hr in air. The XRD of reaction formed powder indicated a well developed LaMnO_3 , a perovskite family. The reaction formed powder was milled in a plastic jar mill with Y-TZP milling media to BET surface area of 15 m²/g. A batch of 30 vol % LaMnO_3 /3Y-TZP was processed using mixtures of LaMnO_3 and Y-TZP (YZ110, SGC&P, Worcester, Mass.) followed by the same preparation method described in Example 1.

Samples were sintered at 1250°–1350° C. to greater than 98% T.D. The density of low temperature (1225° C.) sintered samples was greater than 97% T.D. Sintered samples were HIPed at 1175° and 1350° C. at 207 MPa of argon to full density. The high temperature (1350° C.) sinter-HIPed samples show a significant grain growth, formation of reaction phase, lanthanum zirconate ($\text{La}_2\text{Zr}_2\text{O}_7$), and a few minor surface cracks. The low temperature HIPed samples show a much smaller grain size and no indication of bloating or cracking. Both sintered and HIPed samples were black. It

15 to form lanthanum zirconate ($\text{La}_2\text{Zr}_2\text{O}_7$), which is an insulator. The present results indicate that a tough, black ESD dissipative zirconia can be prepared by mixing LaMnO_3 in Y-TZP by a low temperature sintering.

TABLE 11

ZrO ₂ type	LaMnO ₃ (Vol %)	Densification (Method/Temp.)	R _v (Ohm-cm)
YZ110	20	S/1250° C.*	1.2E11
YZ110	25	S/1250° C.	1.5E08
YZ110	30	S/1250° C.	3.7E07
YZ110	30	H/1175° C.**	6.1E07
YZ110	30	H/1200° C.**	2.6E07
YZ110	30	S/1400° C.	2.8E11
YZ110	30	H/1350° C.**	6.4E12

35 S*: pressureless sintering in air, H**: HIPing in argon

TABLE 12

Property Name	SI Units	(Mean values)	Test method
Color		Light gray	Visual/color meter
Density	g/cm ³	6.13	Water displacement
Porosity	%	less than 0.5%	Density and polished surface
Grain size	µm	1	Linear intercept
Young's Modulus, 20° C.	GPa	182	Ultrasonic wave velocity
Hardness - Vickers, 20° C.	GPa	11.8	Vickers hardness at P = 20 Kgf
Flexural Strength, 20° C.	MPa	716	4-point bending, ASTM-C1161-90
Fracture Toughness, 20° C.	MPa	4.2	Indentation crack length
Fracture Toughness, 20° C.	MPa	3.6	Indentation strength at P = 40 Kgf

is reported in U.S. Pat. No. 6,136,232 (2000) that some perovskite-type compounds such as LaMnO_3 and LaFeO_3 are not chemically stable in combinations with partially stabilized zirconias and at typical sintering temperatures form secondary zirconia compounds such as $\text{La}_2\text{Zr}_2\text{O}_7$, thus effecting the phase stability of the remaining zirconia alloy. The present result indicates that LaMnO_3 can be successfully used to prepare a composite with Y-TZP with a low temperature sintering.

Microstructural observations by SEM from a polished cross section of low temperature sintered sample indicates that the grain size of the material is less than 1 µm. The XRD from a polished cross section also indicated that the majority of tetragonal zirconia was retained with negligible reaction

55 The present result is different from others. For instance, Kyocera's Japanese Patent Application indicates the amount of Mn_2O_3 must be limited to less than 2 Wt. % in order to achieve good zirconia ceramics.

60 The effect of HIPing on the volume resistivity was also determined. Unlike other materials described in Examples 1 and 2, the volume resistivity of the material was not changed before and after sintering. It is believed that various doped LaMnO_3 and LaBO_3 (B Al, Mn or Co)^{19,20} family can be used as semiconductive dispersoids in zirconia.

EXAMPLE 4



65 A batch of LaCrO_3 was prepared by the solid-state reaction of an equimolar powder mixture of La_2O_3 and

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Cr_2O_3 at 1400° C. for 4 hr in air. The XRD of reaction formed powder indicated a well developed LaCrO_3 . The reaction formed powder was milled in a plastic jar mill with Y-TZP milling media to BET surface area of 15 m^2/g . A batch of 30 vol % $\text{LaCrO}_3/3\text{Y-TZP}$ was processes using mixtures of LaMnO_3 and Y-TZP (HSY-3.0, Zirconia Sales of America, Atlanta, Ga.) followed by the same preparation method described in Example 1. Samples were sintered at 1450°–1500° C. in air to greater than 97% T.D. and showed a dark green color with a volume resistivity of 9×10^3 Ohm-cm. Sintered samples were HIPed at 1450° C. at 207 MPa of argon to full density. The color of HIPed samples became slightly lighter than that of sintered only.

Microstructural observations by SEM from a polished cross section indicate that the grain size of the material is about 1 μm . The XRD from a polished cross section also indicated that the majority of tetragonal zirconia was retained with no measurable reaction to form lanthanum zirconate. The present results indicate that a dense, tough, dark colored ESD dissipative zirconia can be prepared by mixing LaCrO_3 in Y-TZP by sinter-HIPing.

TABLE 13

ZrO_2 type	(Vol %)	Temp.)	Densification	Heat treatment	Rv
			LaCrO_3 (Method/		
3.0Y-TZP	10	S/1550° C.			1.0E11
3.0Y-TZP	20	S/1550° C.			3.5E10
3.0Y-TZP	30	S/1550° C.			1.7E07
3.0Y-TZP	40	S/1550° C.			2.0E06
3.0Y-TZP	30	H/1400° C.**			1.2E12
3.0Y-TZP	30	H/1400° C.**	1400 C./1 Hr	Air	5.4E06

S*: pressureless sintering in air, H**: HIPing in argon

TABLE 14

Property Name	SI Units	(Mean values)	Test method
Color		Dark green	Visual/color meter
Density	g/cm^3	6.27	Water displacement
Porosity	%	less than 0.5%	Density and polished surface
Grain size	μm	1	Linear intercept
Young's Modulus, 20° C.	GPa	228	Ultrasonic wave velocity
Hardness - Vickers, 20° C.	GPa	12	Vickers hardness at $P = 20$ Kgf
Flexural Strength, 20° C.	MPa	1020	4-point bending, ASTM-C1161-90
Fracture Toughness, 20° C.	MPa	4.8	Indentation strength (3.0Y-TZP)
Fracture Toughness, 20° C.	MPa	5.7	Indentation strength (2.5Y-TZP)
Fracture Toughness, 20° C.	MPa	8.9	Indentation strength (2.0Y-TZP)
ESD voltage decay time	ms	70	From 1000 V

The resistivities were measured before and after HIPing. Surprisingly, we have found a significant increase, up to 3 orders of magnitude, in resistivity after HIPing. The thermal annealing in air at 1400° C./60 min in air reproducibly recover the resistivity back to the sintered value as summarized in Table 13. The present finding on the reversible resistivity has not been reported in the literature. U.S. Pat. No. 6,136,232 (2000) reports that complex chromate $\text{A}_x\text{B}_y\text{CrO}_3$ can be used to prepare electrostatic dissipative ceramics. Further, it is stated that the material can be HIPed for higher density. However, it was not disclosed that the resistivity of such material drastically changes with HIPing and a need for adjustment of resistivity by the controlled heat treatment.

Batches with varying amounts of Y2O3: 2Y, 2.5Y, and 3Y (mol. %) were prepared using mixtures of HSY3.0 and

DK-1 (0% Y_2O_3 — ZrO_2 , Zirconia Sales of America) to determine the optimum stabilization of zirconia. The fracture toughness of these compositions are listed in Table 8 showing a higher K_{IC} up to 9 MPam^{0.5} of the composite with 2Y-TZP.

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EXAMPLE 5
Zirconium Carbide (ZrC)

Similarly, ZrC can be mixed with Y-TZP to prepare dense, semi-insulative, ESD dissipative ceramics as listed in Table 16. The semi-insulating compositions were prepared by mixing 10 to 20 vol % of ZrC in Y-TZP (HSY3.0). ZrC does not react with ZrO_2 during the hot pressing at 1550° C./20 MPa in argon using graphite dies. The color of hot pressed ZrC was black. The resulting composite is stiffer and harder than ZrO_2 . Accordingly, the semi-insulating materials with ZrC is slightly stiffer and harder than Y-TZP can be prepared with ZrC.

TABLE 15

ZrC	3Y-TZP (Vol. %)	Hot Pressing (Temp./Hr)	Rv ($\Omega\text{-cm}$)
10	90	1550° C./1 H	1.3E12
11.5	88.5	1550° C./1 H	7.0E11
12.5	87.5	1550° C./1 H	5.2E11
13.5	86.5	1550° C./1 H	3.7E09
15	85	1550° C./1 H	5.4E04
20	80	1550° C./1 H	3.7E04
25	75	1550° C./1 H	2.3E00

EXAMPLE 6
Aluminum Nitride (AlN)

This example shows another ceramic material—aluminum nitride—that can be heat-treated to be suitable for

static dissipation. A 12×12×1 mm square of aluminum nitride was procured from SGCP (Carborundum). Surface resistance was measured to be greater than 10^{10} ohms. This is too high to be able to dissipate charge effectively. The sample was then heat treated in argon at 1900° C. for 20 minutes and cooled rapidly to room temperature at a rate more than 10° C./minute. The surface resistance was measured again on both sides. It was about 2×10^9 ohms, which is in the range suitable for dissipating static electricity.

EXAMPLE 7

Complex Oxides— $\text{BaO } 6\text{Fe}_2\text{O}_3$

Complex oxide compounds such as barium hexaferrite ($\text{BaO } 6\text{Fe}_2\text{O}_3$) can also be mixed with Y-TZP (YZ 110) to prepare dense, semi-insulative, ESD dissipative ceramics.

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The semi-insulating compositions were prepared by mixing 25 vol % of BaO 6Fe₂O₃ (BaFe₁₂O₁₉) in Y-ZP (YZ110). The composition can be sintered at 1300°–1400° C. for 1 Hr. in air to greater than 98% TD. The volume resistivity of the sintered disks was 2×10^7 Ohm-cm. Barium hexaferrite is known as a strong magnetic material. The sintered composite was easily attracted by a permanent magnet indicating a good retainability of BaO 6Fe₂O₃ phase in fine zirconia matrix. In summary, one can also prepare a strong magnetic, ESD dissipative, zirconia ceramics for potential applications.

EXAMPLE 8

Color Measurements

Color measurements were made using two separate methods. First, lightness of color was judged using a 16 level gray scale ranging from white to black. Measurements were also made with a Hunterlab Miniscan XLE calorimeter, made by Hunter Associates Laboratory of Reston, Va. The instrument illuminates the sample target with diffuse illumination and observes the reflected light at an 8 degree angle with a spectrophotometer covering the range 400–700 nm. Output from the calorimeter was presented using the CIE 1976 L*a*b* scale, wherein:

- L* measures lightness and varies between 0 and 100 (100 is white/1 is black);
- a* measures redness when positive, gray when zero and greenness when negative;
- b* measures yellowness when positive, gray when zero and blueness when negative.

We noted that the 50% point on the gray scale corresponded to values of L* higher than 50.

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This lowered surface resistance below 1 MΩ, as measured with the same probe. Electrodes were then attached with silver paint to two locations on the ground face of the bar about 7 mm apart along the length of the bar.

The bar with electrodes was loaded into a standard 4-point bending fixture (with supports 40 mm apart on the tension side and 20 mm apart on the compression side) with the electrodes on the compression side.

Load was applied to the bar and the resistance between the electrodes was measured. The dependence of resistance R on compressive strain ε was found to follow the equation $\partial R / \partial \epsilon = 100$. This value is unusually high—for most conductors $\partial R / \partial \epsilon$ is less than 5. Although the present invention is not limited by theory, it is believed that this behavior results from the fact that only just enough conductor has been added to the insulating zirconia ceramic to make it conductive, and that small perturbations to the material therefore have disproportionately large effects on conductivity.

Several applications suggest themselves. Monitoring the resistance of a component made from this class of materials will provide an indication of the stress in the material. For heavily loaded components, this may lead to early detection of incipient failures. Vibrations in the material should produce oscillations in the electrical conductivity and hence may be conveniently detected.

In the example, the sample was deliberately fabricated to have a non-uniform conductivity. This was beneficial because the current was forced to go through the most highly stressed region of the sample, thereby maximizing the response. In other situations—e.g., a bar in pure tension—the stress may be more uniform and, hence, there would be no need to make the conductivity non-uniform.

In the example, grinding the surface of the sample was used to enhance the conductivity of the sample in one local

TABLE 16

CIE Measurement Results						
Sample	Processing	Density (% theoretical)	Resistivity (MΩ-cm)	L*	a*	b*
A	1250C/ 1 hr sinter, 1200C/ 45 min HIP	99.0	0.0012	75.28	-6.07	10.63
B	1250C/ 1 hr sinter, 1200C/ 45 min HIP	99.0	0.0014	69.06	-5.1	2.53
As above, after 4 hrs/580C in air			10.7	86.07	-4.81	15.94
C	1400C/ 1 hr sinter, 1350C/ 45 min HIP	>99%	3	79.61	2.62	48.21
Kyocera (comparative)	(As supplied)	unknown	10	44.03	-0.04	-0.32

The first two samples (A and B) were made from the HSY3.0 zirconia source and sample C from YZ110. The Kyocera sample was a commercially obtained sample.

EXAMPLE 9

Effect of Perturbations on Conductivity

A 50 mm×4 mm×3 mm bar of composition ZrO₂–20%ZnO was prepared by mixing powders of ZrO₂–2.6%Y₂O₃ and ZnO, milling them together, sintering and then hot isostatic pressing, as in the earlier examples.

FIG. 3 illustrates the test apparatus used herein. The bar was heat treated in air in the range 600°–700° C. and the surface resistance of the bar, as measured with a PRF-912 probe (from ProStat Corporation of Bensonville, Ill.) was found to be over 100 MΩ. One side of the bar was ground.

area. This could also be accomplished by locally reducing the conductive phase by a chemical means—atomic hydrogen for example—or by diffusing in an additive that promotes conductivity in the conductive phase (alumina in zinc oxide, for example), or by locally lowering the concentration of a conductivity inhibitor (lithium oxide in zinc oxide reduces conductivity and the lithium concentration in a lithium doped sample might be reduced by heat treatment in an HCl atmosphere).

Another factor influencing conductivity is the temperature of the specimen. In cases where this varies, it may be advantageous to measure the resistance of the specimen in two locations—one stressed and one not stressed and use the

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results from the not-stressed area to correct for the influence of temperature (See FIG. 2). The case of a beam in flexure allows a slightly different approach:

The two resistance sensors are fabricated by locally increasing the conductivity of the material on opposite faces of the beam. If the two resistance sensors are identical, then the ratio of their resistances will be independent of temperature and will be proportional to the bending stress in the beam.

EXAMPLE 10

Low Temperature Degradation (LTD) Resistant ESD Safe Ceramics

Y-TZP has long been known as tougher and stronger than most other ceramics. It's low temperature degradation (LTD) behavior is, though, a significant limitation for some wear resistant applications at low temperature regime (150°–300° C.). The tetragonal zirconia polycrystals (TZP) transform to monoclinic zirconia polycrystals resulting in volume expansion and microcracking at the low temperature regime. Such transformation can be worst in the presence of humidity, especially under equilibrium steam pressure. An accelerated test has been used to determine the LTD behavior of Y-TZPs as described in U.S. Pat. No. 6,069,103, which is hereby incorporated herein by reference.

MOR bars were autoclaved at 200° and 250° C. under equilibrium steam pressure for 48 hours in an autoclave, followed by 4-point bending strength measurement. Table 16 shows flexural strength of 20 vol. % ZnO/YZ110 after the autoclave treatments. The results indicate that the resistance to LTD of 20ZnO/YZ110 is significantly better than that of typical Y-TZP (TZ-3Y, available from Tosoh, Japan). The improved LTD resistance over Y-TZP is desirable using ESD dissipative, zirconia ceramics in degrading environments.

TABLE 17

Flexural strength after autoclave treatment for 48 hours.		
Autoclave temperature (° C.)	Flexural strength (MPa)	
	20 ZnO/YZ110	TZ3Y
20	1050	1507
200	574	474
250	744	590

EXAMPLE 11

Low Particle Generation

ESD safe ceramics are often used in clean room processing environments of magnetic heads, semiconductor components, and IC manufacturing. If ESD safe ceramics shed particles during their use, sensitive electronic components might be damaged. In addition, those contaminated particles must be cleaned, resulting in a costly down time of expensive processing equipment. Further, contaminated particles may contribute for latent or delayed damage in finished products, contributing to costly warranty repairs. Therefore, it is very desirable to develop a material with drastically reduced particle generation. In addition, an improved surface finish process of such material is highly desirable for low particle generation.

Twenty (20) ZnO/YZ 110 samples of disk and square shapes were tumble finished by rolling in a ten sided mill for 72 hours with quartz or silicon carbide media. Some samples

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were lapped or polished to smoother surface finish to determine the effect of surface finish on the particle generation. A white light interference microscope, Zygo NewView 100, was used to measure surface roughness (Ra) after this tumble finish treatment.

Samples of the 20 ZnO/YZ 110 with various surface finishes were tested with a liquid particle counter (LPC). A sample with 3x4x25 mm in size (machined MOR bar) and surface finish of 3800 Å was used as a baseline. Samples were cleaned using a standard procedure used to clean burnish/glide heads for magnetic disk media. A Crest ultrasonic bath was used to dislodge particles from the samples into water (68 kHz for 30 sec.). Liquid particle counts were taken from this water using an LS-200 PMS. The results were normalized by surface area of test sample and reported as number of particles per cm².

The baseline sample with Ra of greater than 3000 Å showed about greater than 600 particles/cm². The treated samples significantly reduced number of particles as a function of surface finish as shown in Table 18. Rougher surfaces typically generate more particles in sliding friction or physical impact applications due to the abrasive nature of the surface and the material used. Decreasing the surface roughness of 20 ZnO/YZ110 by tumble finish, fine grinding, lapping or polishing reduces its potential for particle generation.

TABLE 18

LPC results as a function of surface finish.		
Surface finish	Ra (Å)	LPC (particles/cm ²)
Baseline	greater than 3000	greater than 600
Tumble finish	2250	250
Tumble finish	1500	120
Fine grinding	1000	78
Lapping	500	37
Polishing	20	20

Tumbling is a low cost, industrial process to clean asperities and burrs. Ceramic thread guides are tumble finished to Ra of 100 Å. The present results indicate that ESD dissipative ceramics can be prepared to very low LPC by HIPing and optimum surface finish. For instance ESD dissipative, ceramic tweezers tips can be tumble finished to low I.P.C.

REFERENCES

The following references are cited herein as background information. To the extent necessary for a full understanding of this invention, the disclosures of these references are hereby incorporated herein by reference:

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4. Popp; V. Wuerttenbe, "Tweezers protecting components against electrostatic discharge—has handling surfaces of non-chafing ceramic of high specific surface resistance" DE 3743630 C, Mar. 16, 1989.

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5. "Tape Guides and Method of Manufacturing", JP 3-5363 A, Jan. 11, 1999.—Kyocera.
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17. C.F. Lam, C. Chang, "Decay-Time Characterization of ESD Materials for Use with Magnetoresistive Recording Heads," EOS/ESD Symposium 97, pp. 373–81, 1997.
18. E. A. Bush, U.S. Pat. No. 3,502,597, Mar. 24, 1970.—"Method of Improving the Electrical Conductivity of Sintered Tin Oxide Electrodes".
19. M. J. DeBarr, et al., "Conduction Mechanism in the La(Al,Mn)₃ System," Point Defects and Related Properties of Ceramics, Ed. T. O. Mason, et al, Ceramic Trans. Vol 24, Am. Ceram. Soc., pp. 229–238, 1991.
20. M. S. Islam, M. Cherry and L. J. Winch, "Defect Chemistry of LaBO₃ (B=La, Mn or Co) Perovskite-type Oxides," J. Chem. Soc., Faraday Trans, 92(90) 479–482 (1996).

The present invention has been described in detail, including the preferred embodiments thereof. However, it will be appreciated that those skilled in the art, upon consideration of the present disclosure, may make modifications and/or improvements on this invention and still be within the scope and spirit of this invention as set forth in the following claims.

What is claimed is:

1. An ESD dissipative ceramic component formed by sintering a mixture comprising a base material and one or more resistivity modifiers for a sufficient time and at a sufficient temperature to achieve at least 99 percent of the theoretical density,

wherein the base material comprises a tetragonal zirconia polycrystal (TZP);

wherein the resistivity modifier comprises from about 5 vol. % to 60 vol. % of the base material and is selected from the group consisting of conductive and semiconductive materials or mixtures thereof;

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and wherein the ESD dissipative ceramic component has the following physical properties;

- (a) volume resistivity ranging from 10³–10¹¹ Ohm·cm;
- (b) flexural strength of at least 500 MPa; and
- (c) voltage decay time of less than 500 ms.

2. The ESD dissipative ceramic component of claim 1, wherein the TZP comprises yttria stabilized TZP (Y-TZP) wherein the yttria is present at from about 2.5 to 4.5 mole percent.
3. The ESD dissipative ceramic component of claim 2, wherein the Y-TZP comprises 2.6 mole percent yttria.
4. The ESD dissipative ceramic component of claim 2, wherein the Y-TZP comprises 2.8 mole percent yttria.
5. The ESD dissipative ceramic component of claim 2, wherein the Y-TZP comprises 3 mole percent yttria.
6. The ESD dissipative ceramic component of claim 1, wherein the resistivity modifier comprises from about 10 vol. % to 50 vol. % of the base material.
7. The ESD dissipative ceramic component of claim 1, wherein the resistivity modifier comprises from about 20 vol. % to 40 vol. % of the base material.
8. The ESD dissipative ceramic component of claim 1, wherein the resistivity modifier comprises from about 25 vol. % to 30 vol. % of the base material.
9. The ESD dissipative ceramic component of claim 1, wherein the resistivity modifiers is selected from the group consisting of oxides, carbides, nitrides, oxycarbides, oxynitrides, and oxycarbonitrides.
10. The ESD dissipative ceramic component of claim 1, wherein the flexural strength is at least 600 MPa.
11. The ESD dissipative ceramic component of claim 1, wherein the flexural strength is at least 700 MPa.
12. The ESD dissipative ceramic component of claim 1, wherein the flexural strength is at least 800 MPa.
13. The ESD dissipative ceramic component of claim 1, wherein the flexural strength is at least 900 MPa.
14. The ESD dissipative ceramic component of claim 1, wherein the flexural strength is at least 1000 MPa.
15. The ESD dissipative component of claim 1, where the component has less than 600 particles/cm² in the particle generation test and where the component has an Ra of less than 3000 Å.
16. The ESD dissipative component of claim 15, wherein the surface finish is produced by tumbling.
17. The ESD dissipative component of claim 15, wherein the color of the ceramic is light.
18. The ESD dissipative component of claim 15, wherein the color of the ceramic is dark.
19. An ESD dissipative ceramic component formed by sintering a mixture comprising a base material and one or more resistivity modifiers for a sufficient time and at a sufficient temperature to achieve at least 99.5 percent of the theoretical density;

wherein the base material comprises a tetragonal zirconia polycrystal (TZP);

wherein the resistivity modifier comprises from about 5 vol. % to 60 vol. % of the base material and is selected from the group consisting of conductive and semiconductive materials or mixtures thereof;

and wherein the ESD dissipative ceramic component has the following physical properties;

- (a) volume resistivity ranging from 10⁵–10⁹ Ohm·cm;
- (b) flexural strength of at least 500 MPa; and
- (c) voltage decay time of less than 500 ms.

20. The ESD dissipative ceramic component of claim 19, wherein the TZP comprises yttria stabilized TZP (Y-TZP) wherein the yttria is present at from about 2.5 to 4.5 mole percent.

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21. The ESD dissipative ceramic component of claim 20, wherein the Y-TZP comprises 2.6 mole percent yttria.

22. The ESD dissipative ceramic component of claim 20, wherein the Y-TZP comprises 2.8 mole percent yttria.

23. The ESD dissipative ceramic component of claim 20, wherein the Y-TZP comprises 3 mole percent yttria.

24. The ESD dissipative ceramic component of claim 19, wherein the resistivity modifier comprises from about 10 vol. % to 50 vol. % of the base material.

25. The ESD dissipative ceramic component of claim 19, wherein the resistivity modifier comprises from about 20 vol. % to 40 vol. % of the base material.

26. The ESD dissipative ceramic component of claim 19, wherein the resistivity modifier comprises from about 25 vol. % to 30 vol. % of the base material.

27. The ESD dissipative ceramic component of claim 19, wherein the resistivity modifiers is selected from the group consisting of oxides, carbides, nitrides, oxycarbides, oxynitrides, and oxycarbonitrides.

28. The ESD dissipative ceramic component of claim 19, wherein the flexural strength is at least 600 MPa.

29. The ESD dissipative ceramic component of claim 19, wherein the flexural strength is at least 700 MPa.

30. The ESD dissipative ceramic component of claim 19, wherein the flexural strength is at least 800 MPa.

31. The ESD dissipative ceramic component of claim 19, wherein the flexural strength is at least 900 MPa.

32. The ESD dissipative ceramic component of claim 19, wherein the flexural strength is at least 1000 MPa.

33. The ESD dissipative component of claim 19, where the component has less than 600 particles/cm² in the particle generation test and where the component has an Ra of less than 3000 Å.

34. The ESD dissipative component of claim 33, wherein the surface finish is produced by tumbling.

35. The ESD dissipative component of claim 33, wherein the color of the ceramic is light.

36. The ESD dissipative component of claim 33, wherein the color of the ceramic is dark.

37. An ESD dissipative ceramic component comprising from about 85 to 60 vol. % Y-TZP and from about 15 to 40 vol. % ZnO densified to at least 99% of the theoretical density by primary and secondary heat treatments.

38. The ESD dissipative ceramic component of claim 37, wherein the composition has an L* color measurement of at least 50 using the CIE 1976 L*a*b scale on an LKE colorimeter.

39. An ESD dissipative ceramic component formed of a sintered composition comprising:

a base material comprising tetragonal zirconia; and
a resistivity modifier comprising about 5 vol % to about 60 vol % of the base material, the resistivity modifier selected from conductive and semiconductive materials and mixtures thereof, wherein

the ESD dissipative ceramic component has a density of at least 99% of the theoretical density of the component.

40. The ESD dissipative ceramic component of claim 39, wherein said Y-TZP is present in an amount of about 90 to 50 vol. %, and said resistivity modifier comprises from about 10 to 50 vol. % semi-conductive SnO₂.

41. The ESD dissipative ceramic component of claim 40, wherein the composition has an L* color measurement of at least 50 using the CIE 1976 L*a*b* scale on an LKE colorimeter.

42. The ESD dissipative ceramic component of claim 39, wherein said Y-TZP is present in an amount of about 80 to

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70 vol. %, and said resistivity modifier comprises about 20 to 30 vol. % LaMnO₃.

43. The ESD dissipative ceramic component of claim 39, wherein said Y-TZP is present in an amount of about 90 to 60 vol. % Y-TZP, and said resistivity modifier comprises about 10 to 40 vol. % LaCrO₃.

44. The ESD dissipative ceramic component of claim 39, wherein said Y-TZP is present in an amount of about 90 to 75 vol. %, and said resistivity modifier comprises about 10 to 25 vol. % ZrC.

45. The ESD dissipative ceramic component claim 39, wherein said Y-TZP is present in an amount of about 75 vol. % and, and said resistivity modifier comprises about 25 vol. % BaO 6Fe₂O₃.

46. The ESD dissipative ceramic component of claim 39, wherein the resistivity modifier comprises and semiconductive SnO₂, said ceramic component having the following physical characteristics:

- (a) predetermined decay time; and
- (b) hardness greater than 10 GPa Vickers.

47. The ESD dissipative ceramic component of claim 46, wherein the SnO₂ is present in the range of 20–80% by vol of the initial reagents.

48. The ESD dissipative component, of claim 39, wherein the component is selected from the group consisting of MR (magnetoresistive) head substrates, transfer tools for magnetic head manufacturing, tools, fixtures, and containers for HGA (head gimbal assembly), HSA (head stack assembly), HDD (hard disk drive) assembly, tools, fixtures, and containers for wafer handling, processing, and cleaning, tools, fixtures, and containers for wire bonding, trimming, cutting, pick-and-place nozzles for IC chip handling, nozzles for adhesive and solder dispensing, wafer handling fixtures, end effectors, vacuum chucks, IC handling and test fixtures, tools, fixtures, and containers for ESD sensitive devices, assembly fixtures for IC's and magnetic heads, tweezers, screw drivers, scissors, blades, parts for robotic fingers, tools, fixtures, and containers for electro-optic coating process, tools, fixtures, and containers for reticles (photo mask).

49. An ESD dissipative ceramic component formed by sintering a mixture comprising a base material and one or more resistivity modifiers;

wherein the base material comprises a tetragonal zirconia polycrystal (TZP);

wherein the resistivity modifier comprises from about 5 vol. % to 60 vol. % of the base material and is selected from the group consisting of conductive and semiconductive materials or mixtures thereof;

and wherein the ESD dissipative ceramic component has the following physical properties:

- (a) volume resistivity ranging from 10⁶–10⁹ Ohm-cm;
- (b) flexural strength of at least 400 MPa; and
- (c) voltage decay time of less than 500 ms.

50. The ESD dissipative ceramic component of claim 49, wherein the sintering is conducted in air.

51. The ESD dissipative ceramic component of claim 50, wherein a secondary heat treatment step comprises hot isostatic pressing.

52. The ESD dissipative ceramic component of claim 49, wherein the resistivity modifier is selected from the group consisting of ZnO, SnO₂, ZrC, LaMnO₃, BaO 6Fe₂O₃.

53. An ESD dissipative ceramic component formed of a sintered composition comprising:

- a base material comprising tetragonal zirconia; and
- a resistivity modifier comprising about 5 vol % to about 60 vol % of the base material, the resistivity modifier

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selected from conductive and semiconductive materials and mixtures thereof, wherein the ESD dissipative ceramic component has an L* color measurement of at least 50 using the CIE 1976 L*a*b* scale on an LKE calorimeter, a density not less than 95% of theoretical density, and a volume resistivity within a range of about 10³ to about 10¹¹.

54. The ESD dissipative ceramic component of claim 53, wherein the ESD dissipative ceramic component has the following physical properties:

- (a) volume resistivity ranging from 10⁵–10⁹ Ohm-cm;
- (b) flexural strength of at least 400 MPa; and
- (c) a residual flux density no greater than about 10 Gauss.

55. The nonmagnetic ESD dissipative ceramic component of claim 54, wherein the resistivity modifiers have a molar magnetic susceptibility of less than 10⁻³ ml/mol.

56. The nonmagnetic ESD dissipative ceramic component of claim 55, wherein the resistivity modifiers are selected from the group consisting of ZnO, SnO₂, LaMnO₃, BaO 20 6Fe₂O₃ and mixtures thereof.

57. The ESD dissipative ceramic component of claim 53, wherein the component is a transfer tool for magnetoresistive head manufacturing, wherein the ESD dissipative ceramic transfer tool has a residual flux density no greater than about 10 Gauss.

58. A method of processing a wafer, comprising: providing a tool for processing the wafer, the tool comprising the sintered composition of claim 53; and processing the wafer utilizing the tool.

59. The component of claim 53, wherein said resistivity modifier comprises at least one of ZnO and SnO₂.

60. The component of claim 53, wherein the density is not less than about 98% of theoretical density.

61. The component of claim 53, wherein the density is not less than about 99% of theoretical density.

62. An ESD dissipative ceramic component formed of a sintered composition comprising:

a base material comprising tetragonal zirconia; and
40 a resistivity modifier comprising about 5 vol % to about 60 vol % of the base material, the resistivity modifier

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selected from conductive and semiconductive materials and mixtures thereof, wherein the component has less than 600 particles/cm² in a particle generation test.

63. A method of processing a wafer, comprising: providing a tool for processing the wafer, the tool comprising the sintered composition of claim 62; and processing the wafer utilizing the tool.

64. The component of claim 62, wherein the particle generation test is carried out by subjecting the component to an ultrasonic bath to dislodge particles into water, at a frequency of 68 kHz for 30 seconds.

65. An ESD dissipative ceramic integrated circuit fixture formed of a sintered composition comprising:

15 a base material comprising tetragonal zirconia; and a resistivity modifier comprising about 5 vol % to about 60 vol % of the base material, the resistivity modifier selected from conductive and semiconductive materials and mixtures thereof, wherein the fixture has a density of at least 99% of the theoretical density.

66. The fixture of claim 65, wherein the fixture is selected from the group consisting of handling, test, and assembly fixtures for integrated circuits.

25 67. The fixture of claim 65, wherein the fixture is a test fixture for integrated circuits.

68. An ESD dissipative ceramic integrated circuit fixture formed of a sintered composition comprising:

a base material comprising tetragonal zirconia; and
30 a resistivity modifier comprising about 5 vol % to about 60 vol % of the base material, the resistivity modifier selected from conductive and semiconductive materials and mixtures thereof, wherein the fixture has less than 600 particles/cm² in a particle generation test.

69. The fixture of claim 68, wherein the fixture is selected from the group consisting of handling, test, and assembly fixtures for integrated circuits.

70. The fixture of claim 68, wherein the fixture is a test fixture for integrated circuits.

* * * * *

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JS 44 (Rev. 3/99)

CIVIL COVER SHEET

The JS-44 civil cover sheet and the information contained herein neither replace nor supplement the filing and service of pleadings or other papers as by law, except as provided by local rules of court. This form, approved by the Judicial Conference of the United States in September 1974 is req the use of the Clerk of Court for the purpose of initiating the civil docket sheet. (SEE INSTRUCTIONS ON THE REVERSE OF THE FORM.)

1. (a) PLAINTIFFS

Saint-Gobain Ceramics and Plastics, Inc.
One New Bond Street, Worcester, Massachusetts 01615

(b) County of Residence of First Listed Plaintiff Worcester
(EXCEPT IN U.S. PLAINTIFF CASES)

(C) Attorney's (Firm Name, Address, and Telephone Number)

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DEFENDANTS
IN CLERK'S OFFICE

CoorsTek, Inc.
16000 Table Mountain Parkway, Golden, Colorado 80403

County of Residence of First Listed Defendant Jefferson (Colorado)

(U.S. PLAINTEES ONLY)

NOTE: IN LAND CONDEMNATION CASES, USE THE LOCATION OF THE TRACT OF LAND INVOLVED.

II BASIS OF JURISDICTION (Place an "X" in One Box Only)

<input type="checkbox"/> 1 U.S. Government Plaintiff	<input checked="" type="checkbox"/> 3 Federal Question (U.S. Government Not a Party)
<input type="checkbox"/> 2 U.S. Government Defendant	<input type="checkbox"/> 4 Diversity (Indicate Citizenship of Parties in Item III)

III CITIZENSHIP OF PRINCIPAL PARTIES (PLACE AN "X" IN ONE BOX FOR PLAINTIFF AND ONE BOX FOR DEFENDANT)
(For Diversity Cases Only)

Citizen of This State	PTF	DEF	PTF	DEF
<input type="checkbox"/>	<input type="checkbox"/> 1	<input type="checkbox"/> 1	<input type="checkbox"/>	<input type="checkbox"/> 4
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> 4
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

Incorporated or Principal Place of Business In This State

Citizen of Another State 2 2 Incorporated and Principal Place 5 5 of Business In Another State

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IV. NATURE OF SUIT (Place an "X" in One Box Only)

CONTRACT	TORTS	FORFEITURE/PENALTY	BANKRUPTCY	OTHER STATUTES
<input type="checkbox"/> 110 Insurance	PERSONAL INJURY	PERSONAL INJURY	<input type="checkbox"/> 422 Appeal 28 USC 158	<input type="checkbox"/> 400 State Reapportionment
<input type="checkbox"/> 120 Marine	<input type="checkbox"/> 310 Airplane	<input type="checkbox"/> 362 Personal Injury	<input type="checkbox"/> 423 Withdrawal 28 USC 157	<input type="checkbox"/> 410 Antitrust
<input type="checkbox"/> 130 Miller Act	<input type="checkbox"/> 315 Airplane Product Liability	<input type="checkbox"/> 365 Personal Injury - Product Liability	PROPERTY RIGHTS	<input type="checkbox"/> 430 Banks and Banking
<input type="checkbox"/> 140 Negotiable Instrument	<input type="checkbox"/> 320 Assault, Libel & Slander	<input type="checkbox"/> 368 Asbestos Personal Injury Product Liability	<input type="checkbox"/> 450 Commerce/ICC Rates/etc	
<input type="checkbox"/> 150 Recovery of Overpayment & Enforcement of Judgment	<input type="checkbox"/> 330 Federal Employers' Liability	PERSONAL PROPERTY	<input type="checkbox"/> 460 Deportation	
<input type="checkbox"/> 151 Medicare Act	<input type="checkbox"/> 340 Marine	<input type="checkbox"/> 370 Other Fraud	<input type="checkbox"/> 470 Racketeer Influenced and Corrupt Organizations	
<input type="checkbox"/> 152 Recovery of Defaulted Student Loans (Excl. Veterans)	<input type="checkbox"/> 345 Marine Product Liability	<input type="checkbox"/> 371 Truth in Lending	<input type="checkbox"/> 810 Selective Service	
<input type="checkbox"/> 153 Recovery of Overpayment of Veteran's Benefits	<input type="checkbox"/> 350 Motor Vehicle	<input type="checkbox"/> 380 Other Personal Property Damage	<input type="checkbox"/> 850 Securities/Commodities/ Exchange	
<input type="checkbox"/> 160 Stockholders' Suits	<input type="checkbox"/> 355 Motor Vehicle Product Liability	<input type="checkbox"/> 385 Property Damage Product Liability	<input type="checkbox"/> 875 Customer Challenge 12 USC 3410	
<input type="checkbox"/> 190 Other Contract	<input type="checkbox"/> 360 Other Personal Injury	Labor	<input type="checkbox"/> 891 Agricultural Acts	
<input type="checkbox"/> 195 Contract Product Liability		<input type="checkbox"/> 510 Motions to Vacate Sentence	<input type="checkbox"/> 892 Economic Stabilization Act	
		<input type="checkbox"/> 520 Employment	<input type="checkbox"/> 862 Black Lung (923)	
		<input type="checkbox"/> 530 Housing/ Accommodations	<input type="checkbox"/> 863 DWDC/DIW (405(g))	
		<input type="checkbox"/> 540 Welfare	<input type="checkbox"/> 864 SSID Title XVI	
		<input type="checkbox"/> 550 Other Civil Rights	<input type="checkbox"/> 865 RSI (405(g))	
		SOCIAL SECURITY	FEDERAL TAX SUITS	
		<input type="checkbox"/> 710 Fair Labor Standards Act	<input type="checkbox"/> 870 Taxes (U.S. Plaintiff or Defendant)	<input type="checkbox"/> 900 Appeal of Fee Determination Under Equal Access to Justice
		<input type="checkbox"/> 720 Labor/Mgmt. Relations	<input type="checkbox"/> 871 IRS-Third Party 26 USC 7609	<input type="checkbox"/> 950 Constitutionality of State Statutes
		<input type="checkbox"/> 730 Labor/Mgmt. Reporting & Disclosure Act		<input type="checkbox"/> 890 Other Statutory Actions
		<input type="checkbox"/> 740 Railway Labor Act		
		<input type="checkbox"/> 790 Other Labor Litigation		
		<input type="checkbox"/> 791 Empl. Ret. Inc. Security Act		

(PLACE AN "X" IN ONE BOX ONLY)

V. ORIGIN

1 Original 2 Removed from State Court 3 Remanded from Appellate Court 4 Reinstated or 5 Reopened Transferred from another district (specify) 6 Multidistrict Litigation 7 Magistrate Judgment

Appeal to District Judge from
Magistrate Judgment

VI. CAUSE OF ACTION (Cite the U.S. Civil Statute under which you are filing and write brief statement of cause. Do not cite jurisdictional statutes unless diversity.)

Patent Infringement, 35 U.S.C. s. 101, et seq.

VII REQUESTED IN COMPLAINT:

CHECK IF THIS IS A CLASS ACTION
 UNDER F.R.C.P. 23

DEMAND \$

CHECK YES only if demanded in complaint:
JURY DEMAND: Yes No

VIII RELATED CASE(S) (See instructions):

IF ANY

JUDGE

DOCKET NUMBER

DATE

March 3, 2004

FOR OFFICE USE ONLY

SIGNATURE OF ATTORNEY OF RECORD

RECEIPT # _____ AMOUNT _____ APPLYING IFFP _____ JUDGE _____ MAG. JUDGE _____

UNITED STATES DISTRICT COURT
DISTRICT OF MASSACHUSETTS**04-40034**FEDERAL BUREAU OF INVESTIGATION
U.S. DEPARTMENT OF JUSTICE

1. Title of case (name of first party on each side only) Saint-Gobain Ceramics and Plastics, Inc. v. Cobalt Tek, Inc.

2. Category in which the case belongs based upon the numbered nature of suit code listed on the civil cover sheet. (See local rule 40.1(a)(1)).

I. 160, 410, 470, R.23, REGARDLESS OF NATURE OF SUIT.

II. 195, 368, 400, 440, 441-444, 540, 550, 555, 625, 710, 720, 730, 740, 790, 791, 820*, 830*, 840*, 850, 890, 892-894, 895, 950.

III. 110, 120, 130, 140, 151, 190, 210, 230, 240, 245, 290, 310, 315, 320, 330, 340, 345, 350, 355, 360, 362, 365, 370, 371, 380, 385, 450, 891.

IV. 220, 422, 423, 430, 460, 510, 530, 610, 620, 630, 640, 650, 660, 690, 810, 861-865, 870, 871, 875, 900.

V. 150, 152, 153.

3. Title and number, if any, of related cases. (See local rule 40.1(g)). If more than one prior related case has been filed in this district please indicate the title and number of the first filed case in this court.

None.

4. Has a prior action between the same parties and based on the same claim ever been filed in this court?

YES NO

5. Does the complaint in this case question the constitutionality of an act of congress affecting the public interest? (See 28 USC §2403)

YES NO

If so, is the U.S.A. or an officer, agent or employee of the U.S. a party?

YES NO

6. Is this case required to be heard and determined by a district court of three judges pursuant to title 28 USC §2284?

YES NO

7. Do all of the parties in this action, excluding governmental agencies of the united states and the Commonwealth of Massachusetts ("governmental agencies"), residing in Massachusetts reside in the same division? - (See Local Rule 40.1(d)).

A. If yes, in which division do all of the non-governmental parties reside?

YES NO

Eastern Division Central Division Western Division

B. If no, in which division do the majority of the plaintiffs or the only parties, excluding governmental agencies, residing in Massachusetts reside?

Eastern Division Central Division Western Division

8. If filing a Notice of Removal - are there any motions pending in the state court requiring the attention of this Court? (If yes, submit a separate sheet identifying the motions)

YES NO

(PLEASE TYPE OR PRINT)

ATTORNEY'S NAME Matthew B. Lowrie, Esq./Aaron W. Moore, Esq.

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